FILE 'HOME' ENTERED AT 18:01:41 ON 27 SEP 2004

=> FILE REG

COST IN U.S. DOLLARS

SINCE FILE

TOTAL

ENTRY 0.21 SESSION 0.21

FULL ESTIMATED COST

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=>

Uploading C:\Program Files\Stnexp\Queries\038006.str

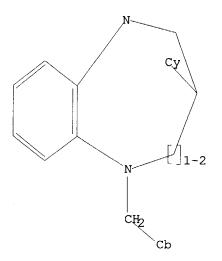
L1 STRUCTURE UPLOADED

=> D L1

L1 HAS NO ANSWERS

L1

STR



Structure attributes must be viewed using STN Express query preparation.

=> S L1 SSS FULL

FULL SEARCH INITIATED 18:02:10 FILE 'REGISTRY' FULL SCREEN SEARCH COMPLETED - 108866 TO ITERATE

100.0% PROCESSED 108866 ITERATIONS

5 ANSWERS

SEARCH TIME: 00.00.02

L2

5 SEA SSS FUL L1

=> FILE CAPLUS

COST IN U.S. DOLLARS

SINCE FILE

TOTAL

FULL ESTIMATED COST

ENTRY 155.42

SESSION 155.63

FILE 'CAPLUS' ENTERED AT 18:02:21 ON 27 SEP 2004

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FILE COVERS 1907 - 27 Sep 2004 VOL 141 ISS 14 FILE LAST UPDATED: 26 Sep 2004 (20040926/ED)

This file contains CAS Registry Numbers for easy and accurate substance identification.

=> S L2

L3

1 L2

=> D L3 IBIB ABS HITSTR

ANSWER 1 OF 1 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1999:234566 CAPLUS

DOCUMENT NUMBER:

131:44796

TITLE:

Solid-Phase Synthesis of 3,4,5-Substituted

1,5-Benzodiazepin-2-ones

AUTHOR(S):

Lee, Jung; Gauthier, Diane; Rivero, Ralph A.

The R.W. Johnson Pharmaceutical Research Institute, CORPORATE SOURCE:

Spring House, PA, 19477, USA

SOURCE:

Journal of Organic Chemistry (1999), 64(9), 3060-3065

CODEN: JOCEAH; ISSN: 0022-3263

PUBLISHER:

American Chemical Society

DOCUMENT TYPE:

Journal

LANGUAGE:

English

OTHER SOURCE(S):

CASREACT 131:44796

The preparation of 3,4,5-substituted 8-carboxamido-1,5-benzodiazepin-2-ones using a solid-phase synthetic method is described. 4-Fluoro-3nitrobenzoic acid is tethered to a solid support via the acid group. Aromatic substitution of the aryl fluoride with either an  $\alpha-$  or  $\beta-$  substituted  $\beta-$  amino ester is carried out in the presence of DIEA in DMF. The reduction of the aryl nitro group is accomplished in the presence of SnCl2·H2O. Hydrolysis of the ester is carried out in the presence of a heterogeneous mixture of 1 N NaOH/THF (1:1). The resulting aniline acid is cyclized to form the benzodiazepinone skeleton with DIC and HOBt. Selective alkylation at the N-5 position of the benzodiazepinone is accomplished with alkyl halides in the presence of K2CO3 in acetone. The desired products are cleaved from solid supports and obtained in 46-98% isolated yields.

IT 224812-11-7P 224812-13-9P 224812-15-1P 224812-17-3P 224812-19-5P

RL: SPN (Synthetic preparation); PREP (Preparation)

(solid-phase synthesis of benzodiazepinones)

RN 224812-11-7 CAPLUS

CN 1H-1,5-Benzodiazepine-7-carboxamide, 2,3,4,5-tetrahydro-4-oxo-3-phenyl-1-(phenylmethyl)- (9CI) (CA INDEX NAME)

$$\begin{array}{c|c} O & H & O \\ \parallel & \parallel & C-NH_2 \\ \hline Ph & N & CH_2-Ph \end{array}$$

RN 224812-13-9 CAPLUS

CN Benzoic acid, 4-[[7-(aminocarbonyl)-2,3,4,5-tetrahydro-4-oxo-3-phenyl-1H-1,5-benzodiazepin-1-yl]methyl]-, methyl ester (9CI) (CA INDEX NAME)

RN 224812-15-1 CAPLUS

CN 1H-1,5-Benzodiazepine-7-carboxamide, 2,3,4,5-tetrahydro-1-[(3-methoxyphenyl)methyl]-4-oxo-3-phenyl- (9CI) (CA INDEX NAME)

10/038,006

RN 224812-17-3 CAPLUS

CN 1H-1,5-Benzodiazepine-7-carboxamide, 2,3,4,5-tetrahydro-1-(2-naphthalenylmethyl)-4-oxo-3-phenyl- (9CI) (CA INDEX NAME)

RN 224812-19-5 CAPLUS

CN 1H-1,5-Benzodiazepine-7-carboxamide, 2,3,4,5-tetrahydro-1-[(4-nitrophenyl)methyl]-4-oxo-3-phenyl- (9CI) (CA INDEX NAME)

REFERENCE COUNT:

THERE ARE 44 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

=> file reg

COST IN U.S. DOLLARS SINCE FILE TOTAL ENTRY SESSION

FULL ESTIMATED COST 5.20 160.83

DISCOUNT AMOUNTS (FOR QUALIFYING ACCOUNTS) SINCE FILE TOTAL ENTRY SESSION

CA SUBSCRIBER PRICE -0.70 -0.70

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=> Uploading C:\Program Files\Stnexp\Queries\038006b.str

L4 STRUCTURE UPLOADED

=>

Uploading C:\Program Files\Stnexp\Queries\038006a.str

L5 STRUCTURE UPLOADED

=> s 14

SAMPLE SEARCH INITIATED 18:10:10 FILE 'REGISTRY' SAMPLE SCREEN SEARCH COMPLETED - 12909 TO ITERATE

7.7% PROCESSED 1000 ITERATIONS INCOMPLETE SEARCH (SYSTEM LIMIT EXCEEDED) SEARCH TIME: 00.00.01

FULL FILE PROJECTIONS: ONLINE \*\*COMPLETE\*\*

BATCH \*\*COMPLETE\*\*

PROJECTED ITERATIONS: 251375 TO 264985 PROJECTED ANSWERS: 0 TO 0

L6 0 SEA SSS SAM L4

=> s 15

SAMPLE SEARCH INITIATED 18:10:16 FILE 'REGISTRY'

0 ANSWERS

SAMPLE SCREEN SEARCH COMPLETED - 14387 TO ITERATE

7.0% PROCESSED 1000 ITERATIONS

INCOMPLETE SEARCH (SYSTEM LIMIT EXCEEDED)

SEARCH TIME: 00.00.01

PROJECTED ANSWERS:

FULL FILE PROJECTIONS: ONLINE \*\*COMPLETE\*\*

BATCH \*\*COMPLETE\*\*

PROJECTED ITERATIONS: 28055

280558 TO 294922 0 TO 0 0 ANSWERS

L7 0 SEA SSS SAM L5

=> file reg

COST IN U.S. DOLLARS SINCE FILE TOTAL

FULL ESTIMATED COST ENTRY SESSION 7.14 167.97

DISCOUNT AMOUNTS (FOR QUALIFYING ACCOUNTS) SINCE FILE TOTAL

CA SUBSCRIBER PRICE ENTRY SESSION -0.70

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STRUCTURE FILE UPDATES: 26 SEP 2004 HIGHEST RN 752189-88-1 DICTIONARY FILE UPDATES: 26 SEP 2004 HIGHEST RN 752189-88-1

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=>
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L8 STRUCTURE UPLOADED

=> Uploading C:\Program Files\Stnexp\Queries\038006c.str

L9 STRUCTURE UPLOADED

=> d 18

L8 HAS NO ANSWERS

L8 STR

10/038,006

G1 O, S, N

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=> d 19

L9 HAS NO ANSWERS

L9 STI

Structure attributes must be viewed using STN Express query preparation.

=> s 18 sss full

FULL SEARCH INITIATED 18:16:56 FILE 'REGISTRY'
FULL SCREEN SEARCH COMPLETED - 8820 TO ITERATE

100.0% PROCESSED 8820 ITERATIONS

684 ANSWERS

SEARCH TIME: 00.00.01

L10 684 SEA SSS FUL L8

=> s 19 sss full

FULL SEARCH INITIATED 18:17:05 FILE 'REGISTRY'
FULL SCREEN SEARCH COMPLETED - 9942 TO ITERATE

100.0% PROCESSED 9942 ITERATIONS

293 ANSWERS

SEARCH TIME: 00.00.01

L11 293 SEA SSS FUL L9

=> file caplus

COST IN U.S. DOLLARS
SINCE FILE TOTAL
ENTRY SESSION
FULL ESTIMATED COST 312.94 480.91

DISCOUNT AMOUNTS (FOR QUALIFYING ACCOUNTS) SINCE FILE TOTAL ENTRY SESSION

obtain CA references citing the substance. The substance formats must be cited first. The CA File predefined formats are: ABS -- Abstract APPS -- Application and Priority Information BIB -- CA Accession Number, plus Bibliographic Data CAN -- CA Accession Number CBIB -- CA Accession Number, plus Bibliographic Data (compressed) IND -- Index Data IPC -- International Patent Classification PATS -- PI, SO STD -- BIB, IPC, and NCL IABS -- ABS, indented, with text labels IBIB -- BIB, indented, with text labels ISTD -- STD format, indented OBIB ---- AN, plus Bibliographic Data (original) OIBIB ----- OBIB, indented with text labels SBIB ----- BIB, no citations SIBIB ----- IBIB, no citations The ALL format gives FIDE BIB ABS IND RE, plus sequence data when it is available. The MAX format is the same as ALL. The IALL format is the same as ALL with BIB ABS and IND indented, with text labels. For additional information, please consult the following help messages: HELP DFIELDS -- To see a complete list of individual display fields. HELP FORMATS -- To see detailed descriptions of the predefined formats. ENTER DISPLAY FORMAT (IDE):end => d l12 122 ibib abs hitstr L12 ANSWER 122 OF 122 CAPLUS COPYRIGHT 2004 ACS on STN 1958:77247 CAPLUS ACCESSION NUMBER: DOCUMENT NUMBER: 52:77247 ORIGINAL REFERENCE NO.: 52:13730b-i,13731a-i,13732a-c TITLE: Cyclic oxo amines. III. Reactions of substituted 1,2,3, 4-tetrahydro-4-oxoquinolines and of 1,6-dioxojulolidines AUTHOR(S): Ittyerah, P. I.; Mann, Frederick G. CORPORATE SOURCE: Univ. Chem. Lab., Cambridge, UK SOURCE: Journal of the Chemical Society, Abstracts (1958) 467-80 CODEN: JCSAAZ; ISSN: 0590-9791 DOCUMENT TYPE: Journal LANGUAGE: Unavailable OTHER SOURCE(S): CASREACT 52:77247 cf. C.A. 52, 5411c. Earlier studies of the reactions of the above 2 types of keto amines were extended. The structures of the derivs. formed by the action of p-ONC6H4NMe2 (I), by bromination, and by dehydrogenation were elucidated. The action of N2H4 and of HNO3 and the application of the Mannich and the Schmidt reaction were also investigated. The properties

of 1,6-dioxoisojuloline (II) were of particular interest.

1,2,3,4-Tetrahydro-4-oxo-1-methylquinoline (III) (0.3 g.) and 0.3 g.

but

p-Me2NC6H4CHO (IV) in 15 cc. alc. containing 0.1 cc. piperidine refluxed 4 hrs. and cooled gave 0.2 g. 3-p-dimethylaminobenzylidene derivative (V), m. 145° (alc.), of III. The use of NaOH instead of piperidine gave only a sticky product in low yield. 1,2,3,4-Tetrahydro-4-oxo-1-phenylquinoline (VI) (0.3 g.) and 0.3 g. IV in alc. containing 0.2 cc. 10% aqueous

NaOH refluxed 1 hr. and set aside overnight gave 0.4 g. 3-p-dimethylaminobenzylidene derivative (VII), m. 148-50° (Me2CO). The use of piperidine instead of NaOH gave only unchanged VI. I (0.3 g.) in 5 cc. alc. added dropwise to 0.3 g. III in 5 cc. alc. diluted with 2 cc. 10% NaOH at 60° gave 3-(p-dimethylaminoanilino)-1,4-dihydro-1-methyl-4oxoquinoline, needles, m.  $215^{\circ}$  (alc.). A similar experiment with VI gave 75% 1-Ph analog, m.  $175^{\circ}$ . VI (0.7 g.), 0.5 g. 2-quinolinecarboxaldehyde, and 0.1 g. KOH in 15 cc. alc. refluxed 2 hrs. gave 1 g. 1-phenyl-3-(2-quinolylmethylene)-4-oxo derivative, m. 205°. III (1.6 g.) in 30 cc. CCl4 shaken 2 hrs. with 1.7 g. N-bromosuccinimide (VIII) gave 1.9 g. 6-bromo-1,2,3,4-tetrahydro-1-methyl-4-oxoquinoline (IX), m. 85° (alc.). IX refluxed 1 hr. in aqueous alc.-KOH gave unchanged material. IX treated with IV in alc. containing piperidine gave the orange 3-(p-dimethylaminobenzylidene) derivative, m. 165°. A similar experiment with I in alc. containing 10% NaOH gave the yellow 3-(p-Me2NC6H4NH) derivative, m. 281°. VI similarly treated gave the 6-Br derivative (X), m. 95-100° (alc.), unaffected by refluxing aqueous KOH. X gave the yellow 3-(p-Me2NC6H4NH) derivative, m. 210° (alc.). With 3.4 g. VIII and 5 mg. Bz202 shaken 4 hrs. and left overnight was obtained 2.8 g. of the 6,8-dibromo-1-methyl-4-oxo derivative (XI), m. 126°. XI gave the 3-(p-dimethylaminobenzylidene) product, plates, m. 190° (C6H6 or MeOH), and a yellow 3-(p-Me2NC6H4NH) derivative, m. 225°. III (1 g.), 0.1 g. 10% Pd-C, and 10 cc. (CH2OH)2 refluxed 0.5 hr. and the product treated with picric acid gave the picrate (XII) of 1,4-dihydro-1-methyl-4oxyquinoline, m. 226°. Repeating the above experiment but using X gave Similarly, VI gave the picrate of the 1-Ph derivative, m. 136° (alc.). III (0.8 g.) 0.6 g. Me2NH.HCl, 0.2 g. paraformaldehyde, and 15 cc. Me2CHCH2CH2OH refluxed 15 min. and the clear solution treated with 0.3 g. more aldehyde and refluxed a further 0.5 hr. gave 0.8 g. 1,4-dihydro-1,3-dimethyl-4-oxoquinoline, m. 320° (decomposition), sparingly soluble in hot alc., Me2CO, and C6H6. Repetition of this experiment

using 1.1 g. VI gave 1 g. 1,4-dihydro-3-methyl-4-oxo-1-phenylquinoline (XIII), darkened at 295° and slowly decomposed at 320-40°, but immersed at 310° it m. 325°. Thus the initial product in this reaction is the tertiary amine-HCl, which breaks off the dialkylamine-HCl to form the 3-methylene derivative, which then isomerizes to the stable XIII. III (0.8 g.) and 0.12 g. pure N2H4.H2O in 10 cc. alc. containing 0.1 cc. AcOH refluxed 1 hr. gave 0.75 g. N,N'-bis(1,2,3,4tetrahydro-1-methyl-4-quinolylidene)azine (XIV), plates, m. 198° (C6H6). VI in the above experiment gave 73% of the 1-Ph analog, m. 208° (dioxane). XIV in alc. added to a large excess of alc. picric acid deposited the monopicrate, red needles, m. 165° (decomposition). XIV in alc. chilled to 0° and treated with dry HCl gave the red HCl salt. A portion of the red solution treated, before separation of the red crystals, dropwise with cold concentrated HCl gave N2H4.2HCl, m. 198-200°. Alternatively, a fine suspension of XIV in Et20 similarly treated with HCl gave the HCl salt, m.  $85-7^{\circ}$ ; analysis indicated that this was a di-HCl salt. HCl passed 0.5 hr. through molten XIV, initially at 200° and then at 190-200°, gave the di-HCl salt of s-bis(1,2-dihydro-1-methyl-4-quinolyl)hydrazine, m. 295° (decomposition); dipicrate, m. 243° (decomposition) (alc.). Concentrated H2SO4 (8

cc.) added dropwise to 1.6 g. III in 10 cc. CHCl3, the solution stirred a

further 15 min., diluted with 45 cc. cold H2O, neutralized, and extracted with Et20 gave 1 g.  $\beta$ -[N-(o-aminophenyl)-N-methylamino]propionic lactam, needles, m. 170° (C6H6). VI similarly treated gave 91% β-[N-(o-aminophenyl)anilino]propionic lactam, m. 221° (alc. then C6H6). III (1 g.) in 10 cc. AcOH treated below 15° with a mixture of 1 cc. concentrated HNO3 and 10 cc. AcOH, set aside 20 min., and poured into H2O gave 1.2 g. 6-nitro derivative, needles, m. 169° (alc.); phenylhydrazone, red plates, m. 198° (decomposition) (alc.); 3-(p-dimethylaminobenzylidene) derivative, red plates, m. 210° (alc.). p-ClC6H4NH2 on cyanoethylation gave a low yield of p-ClC6H4N(CH2CH2CN)2, which in turn on cyclization gave a small amount of 8-chloro-1,6dioxojulolidine (XIVa). This difficulty also applies to the Br analog, but p-BrC6H4N(CH2CH2CN)2 (XV) and 8-bromo-1,6-dioxojulolidine (XVa) can be readily prepared by direct bromination with IX. p-BrC6H4NH2 (17 g.), 12 g. CH2:CHCN, 12 g. AcOH, and 1.7 g. CuCl refluxed 12 hrs. and poured into 100 cc. NH3 gave p-BrC6H4NHCH2CH2CN, b5 160°, m. 96-8°; the residue in the flask decomposed at higher temps. This experiment when repeated by refluxing 20 hrs. gave 2 g. above compound and 4 g. XV, m. 94-5° (alc.). PhN(CH2CH2CN)2 treated 4 hrs. in CCl4 with 1 mole IX and 0.05 mole Bz202 gave 82% XV. XV heated with AlCl3 in PhCl gave what was apparently 1-(2-cyanoethyl)-1,2,3,4-tetrahydro-4-oxoquinoline, m. 79-81°; phenylhydrazone, m. 250°; 2,4dinitrophenylhydrazone, m. above 360°. Thus under the conditions used for this monocyclization the nuclear Br was removed. II, 0.6 g. 2-quinolinecarboxaldehyde, 15 cc. alc., and 0.1 g. KOH refluxed 1 hr. gave 0.3 g. 1,6-dioxo-2,5-bis(2-quinolylmethylene)julodidine, m. 185° (C6H6). The 7,9-di-Me derivative (XVI) of II similarly treated gave the 7,9-di-Me homolog, m. 225° (C6H6). XVI treated with 2.2 moles IV in refluxing alkali and alc. gave 93% 2,5-bis(p-dimethylaminobenzylidene)-7,9-dimethyl-1,6-dioxojulolidine, needles, m. 245° (alc.). XVI similarly treated with 2.1 g. I gave 83% 2-(p-dimethylaminoanilino)-5-)pdimethylaminophenylimino) - 7,9-dimethyl-1,6-dioxojulolidine (XVII), purple crystals, m. 268° (decomposition) (Me2CO). XVII (0.5 g.) refluxed 0.5 hr. with 20 cc. concentrated HCl gave 1.25 g. 7,9-dimethyl-1,2,5,6tetraoxojulolidine (XVIII), bronze-colored crystals, slowly decomposed at 330°. XVIII resembles the parent member in that it is insol. in all the usual solvents, but soluble in aqueous KOH to give a purple solution It did

not condense with p-C6H4(NH2)2. IX (2 g.) and 0.05 g. Bz2O2 shaken 3 hrs. with 1 g. II in CCl4 gave 1.2 g. XVa, plates, m. 208°,  $\lambda$ 421.5-25.5, 301-2.5, 227, 338.5-40, and 276.5 m $\mu$ ,  $\epsilon$  5130, 4490, 30,100, 252, and 1650. When the mixture was refluxed only a brown amorphous intractable material was obtained. XVa gave a bis(phenylhydrazone), m.  $270\,^{\circ}.~$  XVa (0.28 g.) and 0.22 g. BzH in 15 cc. alc. and 0.1 cc. piperidine refluxed gave 0.2 g. 2,5-dibenzylidene-8-bromo-1,6dioxojulolidine, m. 200° (alc.). II treated with I mole N-chlorosuccinimide under the above conditions at room temperature gave no reaction, but warming the mixture under reflux 1 hr. gave 68% XIVa, m. 201°. II (1 g.), 0.1 g. 10% Pd-C, and 25 cc. p-cymene refluxed 1.5 hrs., a slow stream of CO2 blown through the solution, and the hot mixture filtered and cooled gave 0.6 g. 1,6-dioxoisojuloline (XIX), m. 202° (lower yields were obtained in expts. on a larger scale); HBr salt, m. 270° (decomposition); picrate, m. 202°; diperchlorate, m. 240° (decomposition). A cold aqueous suspension of this salt with NH4OH gave XIX. An alc. solution of the salt treated overnight at 0° with excess HClO4 gave the normal perchlorate, m. 125°, and these crystals dissolved in hot alc. gave the diperchlorate on cooling; metho-p-toluenesulfonate, m. 180° (decomposition); phenylhydrazone-0.5H2O, plates, m. 220°. 2-(p-Dimethylaminobenzylidene)-1,6dioxoisojulolidine prepared in refluxing alc. with piperidine formed red

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crystals, m. 230° (dioxane). Although the conditions of the above reduction were varied over wide limits no further dehydrogenation could be effected. XIX (0.33 g.), 0.22 g. CH2(CN)2, 0.1 g. NH4OAc, 10 cc. C6H6, and 1 cc. AcOH refluxed 45 min. gave 1-(dicyanomethylene)-6-oxoisojulolidine, needles, m. 245° (decomposition). Attempted application of the Mannich reaction to II under the above conditions gave solely an intractable powder of indefinite m.p. II (2 moles), 1 mole N2H4.H2O, and a few drops of AcOH on heating gave a complex orange polymeric mixture, m. 280-320°. II gave a deep yellow mixture XVI (1 g.), 1 g. 100% N2H4.H2O, 0.5 g. KOH, and (CH2OH)2 refluxed 2 hrs., heated 4 hrs. at 195-200°, the mixture diluted with H2O, cooled, the solids collected, extracted with alc., and the exts. evaporated gave with picric acid

picrate, almost certainly julolidine picrate. XVI similarly treated gave the 7,9-dimethyljulolidine picrate, m. 160° (alc.). II in CHCl3 with H2SO4 and NaN3 gave 81% dilactam of 2,6-diamino-N,N-bis(2-carboxyethyl)aniline, plates, m. 356° (decomposition) (AcOH). 7-Methyl-1,6-dioxojulolidine (2.1 g.), 3.1 g. isatin, and 3.6 g. KOH in 25 cc. MeOH and 5 cc. H2O refluxed 20 hrs. gave 3 g. 7-methyl-6-oxoquinolino(2',3'-1,2)juloline-4'-carboxylic acid (XX), m. 215° (decomposition). XX heated in a tube at 210-20°/0.1 mm. underwent decarboxylation and gave 7-methyl-6-oxoquinolino(2',3'-1,2)juloline, plates, m. 185°, unchanged by further crystallization or sublimation. II (1 g.) in 10 cc. AcOH treated with 1 cc. concentrated HNO3 below 20° and set aside 20 min. gave 1.2 g. 8-nitro-1,6-dioxojulolidine, m. 250°; bis(phenylhydrazone), m. 270° (decomposition).

32900-17-7, 2H-1,5-Benzodiazepin-2-one, 1,3,4,5-tetrahydro-5-phenyl- 33035-62-0, 2H-1,5-Benzodiazepin-2-one, 1,3,4,5-tetrahydro-5-methyl-

(preparation of) 32900-17-7 CAPLUS

CN 2H-1,5-Benzodiazepin-2-one, 1,3,4,5-tetrahydro-5-phenyl- (6CI, 8CI, 9CI) (CA INDEX NAME)

RN 33035-62-0 CAPLUS CN 2H-1,5-Benzodiazepin-2-one, 1,3,4,5-tetrahydro-5-methyl- (6CI, 8CI, 9CI) (CA INDEX NAME)